“SYNTHESIS, MODIFICATION AND CHARACTERIZATION OF MESOPOROUS METALLOSILICATE MATERIALS WITH THEIR PHYSICAL AND CATALYTIC ACTIVITY”

Pre-Synopsis

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PRE SYNOPSIS

"SYNTHESIS, MODIFICATION AND CHARACTERIZATION OF MESOPOROUS METALLOSILICATE MATERIALS WITH THEIR PHYSICAL AND CATALYTIC ACTIVITY"

1.1. Introduction to Mesoporous Silica:

Mesoporous silica is a kind of silica material that has pores with diameters ranging from 2 to 50 nanometers. These materials possess significant surface areas, a limited range of pore sizes, and a substantial volume of pores. Common instances are SBA-36, SBA-15, and KIT-6.

Mesoporous silica is produced by sol-gel techniques and template-assisted methods, which result in the production of a mesoporous structure [1,2].

Catalysis and Separation:

Mesoporous silica materials are widely esteemed in catalytic applications because of their elevated surface area and consistent pore structure. Consequently, this provides an ideal environment for catalytic reactions. They have the ability to function either as supports for metal catalysts or as catalysts themselves. Furthermore, their adjustable pore diameters render them suitable for separation procedures such as the selective adsorption of molecules[3-5].

- **Drug Delivery:**

Mesoporous silica is used in medication delivery systems within the pharmaceutical business. One advantage is that it offers a large surface area to hold a significant amount of drugs, while also having a porous structure that allows for regulated and prolonged release of the active therapeutic agents. Functionalising the surface of mesoporous silica enhances its ability to selectively interact with certain cells or tissues .

- **Sensors and Detection:**

Sensors are developed using mesoporous silica materials. This is due to their extensive surface area and strong reactivity, which enable them to be modified with chemical groups for the purpose of detecting certain analyses or environmental changes. Therefore, they are valuable in the field of environmental monitoring and medical diagnostics.

❖ **Nanotechnology and Material Science:**

Mesoporous silica plays an essential role in the fields of nanotechnology and material science. It is used in the production of nanostructured materials and composites, with applications ranging from nano capsules to nano carriers, since it can be customised to contain nanoscale substances. Mesoporous silica is a versatile class of materials that has a broad range of uses in both scientific and industrial domains. Indeed, the reason for this is specifically attributed to the amalgamation of distinctive structural characteristics with versatile functionality[7-9].

1.1.1. Synthesis of ordered mesoporous silica

Producing structured mesoporous silica from agro-industrial waste is a sustainable method for transforming trash into a valuable commodity. This process use waste materials either as a source of silica or as a template in the creation of mesoporous silica. As a result, it offers benefits in terms of both environmental sustainability and industrial applications .

The techniques used include direct extraction and silica utilisation, wherein agricultural and industrial wastes are gathered based on their silica content. Examples of such wastes are rice husks, bagasse, and fly ash. The materials undergo chemical treatment to extract silica. For instance, rice husk ash is treated with an alkaline solution to dissolve any non-silica components. The silica that has been extracted is dissolved in a solvent to create a solution of silica precursor. Additional processing may be carried out using similar techniques, sometimes with the assistance of surfactants that serve as structure-directing agents to produce ordered mesoporous silica[10-12].

Ultimately, the methods for obtaining silica from biomass include using organic materials that are abundant in silica, such as rice husks or maize stover. The biomass materials may be either incinerated or subjected to chemical treatment to provide silica, which can then be used in various traditional synthesis techniques such as sol-gel processing, template-assisted synthesis, or hydrothermal procedures to generate mesoporous silica.

These synthesis techniques highlight an eco-friendly approach to producing ordered mesoporous silica by converting waste materials into valuable substances. This significantly contributes to sustainability in the field of materials science.

1.1.2. Application of mesoporous silica

Mesoporous silica materials have garnered significant scientific attention for their distinctive properties, making them highly sought after for a range of applications. The material has certain

structural characteristics, such as a consistent and adjustable pore size ranging from 2 to 6 nanometers, a consistent and adjustable shape, a high surface area of 700 to 1,000 square meters per gram, a large pore volume exceeding 0.9 cubic centimeters per gram, biocompatibility, and the potential for surface modification. Due to the aforementioned distinctive characteristics, these materials are well-suited for many applications, particularly in adsorption and release functions in medication delivery, environmental cleanup, catalytic supports, energy conversion and storage, and electrochemical sensors.

Enhancing the dissolution of a weakly water-soluble medication may be achieved by increasing its effective surface area when it comes into contact with the dissolving media. One way to do this is by loading pharmaceuticals onto ordered mesoporous silica materials. This allows for consistent and uniform drug loading and release with the use of an external triggering factor such as pH, temperature, redox potential, or light irradiation.

1.1.3. Significance of Using Industrial and Agro-Waste:

Utilizing industrial and agro-waste for synthesizing ordered mesoporous silica is a green method of immense applicability. The following are some merits that make it important:

- **Sustainable Environment:**
- **Cost-Effectiveness:**
- **Material Properties Enhancement:**
- **Economic Development and Innovation:**
- **Applications and Versatility:**
- **High Potential Applications**.
- **Toward Technological Application for Sustainabili**ty.

1.1.4. Environmental and economic benefits of using wastes

Utilization of wastes from industries and agro-products in the ordered mesoporous silica synthesis presents a huge environmental and economic benefits, hence a sustainable approach with wide-ranging socio-economic positive impacts.

- **Waste Reduction and Management:**
- **Resource Conservation:**

- **Greenhouse Gas Emission Reduction:**
- **Economic Benefits:**
- **Low-Cost Raw Materials:**
- **Value Addition to Waste Materials:**
- **Job Creation and Economic Growth:**
- **Circular Economy Support:**

1.1.5. Sources and general information on types of used industrial and agro-waste.

The industrial and agro-waste use a wide array of waste materials either as sources of silica or as templates to synthesize ordered mesoporous silica. These materials are sourced according to the silica content in them, availability, and other properties they can infuse into the resultant mesoporous silica[13-15].

- **Sources of Industrial Waste**
- **Fly Ash**

It is the by-product of coal combustion in power plants. It contains the required content of silica and alumina, therefore it is one such source for the extraction of silica. Fly ash is readily available and can be processed to extract silica at a low cost, and this silica will be used in the mesoporous silica synthesis. Using a fly ash will represent a sustainable source of silica but also help to mitigate environmental pollution associated with it disposal[16].

- **Rice husk ash (RHA):**

RHA is a by-product obtained through the rice husk burning that could often be found after rice mills. RHA is one of the sources rich in amorphous silica and is found to be a potential candidate for synthesizing mesoporous silica. Silica derived from RHA finds applications in catalysis as well as environmental remediation. Utilization of RHA will also solve the environmental problem related to its disposal.

- **Bagasse Ash:**

Bagasse ash is the byproduct collected from the incineration of bagasse, the fibrous residue remaining after sugar cane is crushed to extract juice. The most abundant constituent of Bagasse ash is silica, which makes it an attractive choice in synthesizing mesoporous silica materials. The

process adds value to the otherwise waste and also helps to manage the enormous amount of Bagasse ash generated through the sugar industries[17,18].

- **Slag:**

Slag is the by-product that arises during the metal smelting and refining operations, primarily from Steel industries. Some slags are considered as silica abundant materials and suitable for further processing for separation of silica. The extracted silica is then employed for the synthesis of mesoporous silica. In this way, mesoporous silica can be prepared from the by-product slag. The common source is agro waste, which does not only reduce environmental pollution from the ash and thus promote recycling of industrial by-products but also serves as mesoporous silica preparation..

- **Hybrid Catalysts:**

Hybrid biophysico catalysts involve biological components associated with physical or chemical materials to create multifunctional catalytic systems. The hybrids, therefore follow both the benefits. Accruable from the biological and physical processes.An hybrids increase catalytic efficiency in chemical synthesis and environmental applications[19-21].

1.1.6. Sources of the used industrial and agro-waste

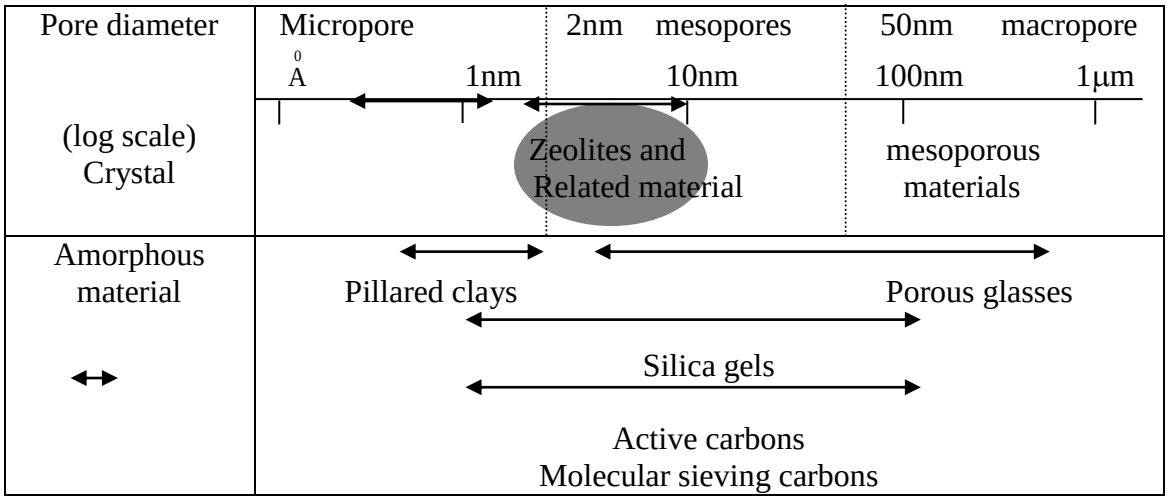
The surfactant micelle conformation, silica-surfactant interactions, and degree of silica poly condensation are influenced by several synthesis variables, including the supply of silica, type of surfactant, ionic strength, pH and content of the reaction fluid, as well as the temperature and time of synthesis. The properties of the porous structure, such as the kind of mesostructure, diameter and volume of the pores, and wall thickness, are determined by these circumstances. Additionally, these conditions also impact the macroscopic morphology.

2. REVIEW OF LITERATURE AND DEVELOPMENT IN THE SUBJECT:

Pore Size (nm)

Microporous : < 200 Mesoporous : <200-5000 Macroporous : > 5000

Table - 1 : Classification of porous materials[20]



In recent years, green concepts and economic thoughts have played a major and growing role of interest to find creative ways not only to reduce the natural wastes and industrial hazard to overcome the environmental pollution but also in redesigning commercially important processes so as to avoid the use of detrimental substances and the generation of venomous waste.

Among all the types of wastes, for the effective utilization of industrial wastes and agricultural wastes, we have opted these wastes produced after pyrolyzing coal and rice husk, sugar cane husk or pea nut husk at elevated temperature as the raw material and feed stock in our further studies rather than depleting them.

Coal Fly ash (CFA), Rice husk ash (RHA), Sugar cane husk (SCH) or Pea nut husk (PNH) are the low cost and readily available silica rich raw materials which can be used for the preparation of value added porous materials like zeolite molecular sieves. Converting these wastes into porous materials not only eliminates the disposal problems, but also turns an otherwise waste material into a marketable commodity as they have widespread applications in diversified fields.

The major components of CFA, RHA, SCH or PNH are silicon dioxide (SiO_2) and Aluminium trioxide (Al_2O_3), which are the essential reagents in the synthesis of zeolites (Shigemoto et.al., 1993; Chang and Shish,1998). Synthetic zeolites obtained by reprocessing of CFA and RHA, sugar cane husk (SCH) or pea nut husk (PNH) have huge potential as a cost-effective,

environmentally-friendly solution that can improve the efficiency of these materials as efficient heterogeneous catalysts, mainly as solid acids, as adsorbents, as molecular sieves in gas separation and purification and in purification of contaminated waste water.

'Zeolites' the aluminosilicate molecular sieves are microporous materials which are inorganic composites. They have a crystalline structure woven with tetrahedral atoms like Al, P, Si etc bridged by oxygen atoms acquiring regular channels or cavities with an explicit number of tetrahedral atoms. The crystallographic unit cell of the zeolites may be represented as:

$$\mathbf{M_{x/n}[(AlO_2)_x(SiO_2)y]\ zH_2O}$$

Where,

M is a charge compensating cation with valency 'n'.

The ratio 'x/y' may have any value ranging from zero to one. The number of water molecules, which can be reversibly adsorbed and desorbed in the zeolite pores, is represented by 'z'. Total number of tetrahedral sites in the unit cell of zeolites represented by x + y.

In general, the ratio (y/x > 1) controls the morphology of zeolites and acidity to a lesser extent.

A number of models such as Liquid Crystal Templating (LCT) Mechanism, Silicate Rod Assembly, Charge Density Matching and Folding Sheets etc. have been proposed in order to explain the mechanism of formation of mesoporous materials by various synthesis routes depicted in fig.1.

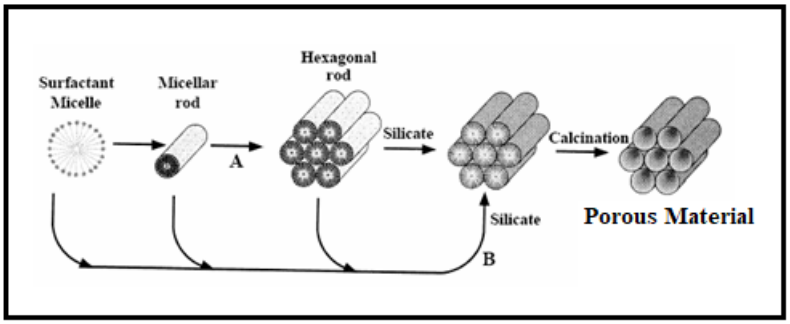


Fig.1: Liquid crystal templating mechanism proposed for the formation of Mesoporous Materials : (A) liquid crystal phase initiated and (B) silicate anion initiated.

All these models are based on the presence and the role of surfactants in solution to guide the formation of inorganic mesostructures. In solution, surfactants having two parts within the same molecule (hydrophilic head group and long chain hydrophobic tail group) will aggregate and self

organize in such a way as to minimize contact between the incompatible ends. The type of interaction between the surfactant and the inorganic precursor under different synthesis conditions needs careful attention and is a subject of much discussion.

In order to generate potential catalytic activities, the incorporation of heteroatoms into the inert framework or walls of pure siliceous mesoporous materials is an important route to modify the nature of the framework and make them catalytically active.

In general, it is observed that the incorporation of tetrahedrally coordinated Aluminium on the walls during the synthesis decreases the order in the material and the degree of incorporation depends on the Aluminium precursor and method of preparation. The incorporation of trivalent metal ions such as Al, B, Ga, Fe etc. in the walls of silica network mesostructure produces framework negative charges that can be compensated by protons providing acid sites and therefore, such materials are important from the point of view of acid catalysis.

The incorporation of transition metal elements such as Ti, V, Cr, Mn, Fe, etc. is also important to prepare mesoporous catalysts with redox catalytic properties. The incorporation of alkali metal elements such as Na, K, Rb and Cs are also important to prepare mesoporous catalysts with basic catalytic properties.

MESOPOROUS SILICATES AS SUPPORT/HOST:

Immobilization of Metals/Metal Complexes in Mesoporous Silicates:

The post-synthesis immobilization of metals and organometallic complexes on the surface of mesoporous silicates allows the preparation of multifunctional molecular sieves with desired catalytic properties. Mesoporosity and very high surface area of mesoporous materials have been largely used for the immobilization of different metals as well as bulky organometallic complexes. The processes for metal immobilization include wet impregnation, vapor deposition, treatment with $(NH_4)_2MF_6$ (M = Si4+, Ti4+, etc.) and metal alkoxides, ion exchange with metal salts etc.

The immobilization of metal complex is mostly done by ion exchange followed by the complexion with ligand and also by direct incorporation of the metal complex in the mesoporous hexagonal channels. The advantages of inorganic-organic hybrid materials arise from the fact that inorganic components can provide mechanical, thermal or structural stability, while the organic features are more readily modified for specific applications in catalysis, separation or sensing.

The presence of large amount of solano groups [(-O-)$_3$SiOH] in mesoporous materials attracted the researchers primarily for anchoring the organic functional groups using the concept of organic function alization in silica gel and then those organic functional groups or ligands were used for anchoring different types of metal complexes with or without modification of the parent functional groups.

Organically functionalized mesoporous silica were prepared conveniently at room temperature or at higher temperature by the co-condensation of tetraalkoxysilane (Si(OR)$_4$) and organosiloxanes (R1□Si(OR)$_3$) in the presence of surfactant (template) and auxiliary chemicals. Thus, a variety of inorganic -organic hybrid materials, where the organic functional groups are attached covalently with the silica surface of porous materials through Si-C bonds, are very important for their various potential applications. An acidic solvent extraction technique is generally used to remove the surfactant from the product to yield an organo functionalized ordered porous silica material.

Organo functional groups were also introduced to the pore surface of mesoporous silica as the terminal groups for organic monolayers by post synthesis modification. The treatment of mesoporous silica with organo siloxane precursors produces hybrid inorganic-organic materials by hydrolysis and finally condensation of organo siloxane groups.

Although the organic functional groups have been introduced into mesoporous silica in both ways, *in situ* and post synthetic methods, organometallic complexes attached covalently to the pore surface are introduced only by post synthesis modifications. Extensive research efforts have been undertaken to control the crystal size, pore dimensions and pore sizes. The control of crystal size and pore dimensions are also important factors, particularly for SBA-36, 48 with unidirectional channels. High quality hexagonal mesoporous materials of good thermal stability and smaller crystal sizes were obtained by heating precursor gels in the microwave at 150ºC for 1 h or even less.

PHYSICO-CHEMICAL CHARACTERIZATION:

Various techniques are used to characterize mesoporous molecular sieves. Among them,

1. X-ray powder diffraction for Phase identification (Qualitative analysis), Quantitative phase analysis, Determination of crystallinity, Crystallite size
2. Adsorption measurements,
3. Scanning electron microscopy (SEM) are the indispensable ones to recognize the configuration of the mesoporous molecular sieves

4. Diffuse reflectance U.V. spectroscopy and Infrared spectroscopy have also been applied to obtain additional structural information about molecular sieves.
5. TG-DTA is performed to examine the thermal stability of the synthesized material.
6. Surface area (BET) and Pore size distributions (BJH) and pore volume and pore size distribution by the BJH Method.
7. Fourier transforms infrared (FTIR) spectroscopy
8. Atomic absorption spectroscopy (AAS)
9. TPD of ammonia
10. Energy Dispersive Analysis of X-ray (EDAX)
11. Gas Chromatography Mass Spectrometry (GC-MS)
12. X-ray fluorescence Spectroscopy (XRF)

APPLICATIONS OF MESOPOROUS MATERIALS:

Waste water treatment methods:

Some of the common treatment processes for purification of waste water are:

* Chemical precipitation,
* Coagulation–precipitation,
* Membrane filtration,
* Reverse osmosis and
* Adsorption and ion exchange

Among the various types of purification methods, ion exchange methodology is the cost effective methodology and is one of the most well-developed, common, and effective treatment methods for treatment of contaminated water. Mesoporous materials are the well-known for its ion exchange capacity. Ion exchange method has been proved to be very effective for removing contaminants from water and increase the water quality.

3. OBJECTIVES OF THE RESEARCH / PROPOSED HYPOTHESIS

The objectives of the present work are:

1. Extraction of silica from CFA, RHA, CSH and PNH

2. To synthesize SBA-36 using silica extracted from CFA and RHA, CSH, PNH and to optimize the synthesis procedures.

3. To study the kinetics of crystallization on synthesized materials such as

   - Effect of crystallization time
   - Effect of Temperature
   - Effect of P^H
   - Effect of calcination period
   - Effect of NaOH

4. Post Modification of synthesized materials (metal incorporated synthesized materials).

5. To characterize the synthesized samples and modified samples of synthesized materials in detail using various techniques and tools such as elemental analysis, XRD, N_2-adsorption measurements, electron microscopy (SEM), TGA/DTA, ammonia-TPD, UV-visible, FT-IR, etc.

6. To study the effect on the adsorptive behavior of pre and post modified synthesized materials

7. To study the catalytic activity of synthesized materials in diverse reactions.

8. To deal with an account of conductivity behavior of selected samples.

4. METHODOLOGY TO BE ADOPTED:

Hydrothermal synthesis of mesoporous molecular sieves was carried out in hydrothermal reactor at autogenously pressure under stirring conditions. However an ion exchange method will be adopted for post modification and to perform some heterogeneous chemical reactions and also to study some 'Freundlich adsorption' applications

5. IMPORTANCE OF STUDY:

Synthesis of mesoporous materials from chemical sources of silica and alumina is expensive therefore; their use in environmental remediation is restricted due to prohibitive costs, which can be overcome by seeking cheaper raw materials for their synthesis. These include clay minerals, rice husk ash, coal fly ash, etc.

Therefore, our research endeavors the use of no-cost raw materials such as industrial waste CFA collected from the Thermal Power Stations and agro waste rice husk from the rice mills. The reasons for choosing these sources for our further investigations are:

1) They add value; reduce the cost and creates effective utilization to protect the environment.

2) Their potential application in synthesis of highly useful porous materials.

3) Their potential application in water purification technology, as industrial catalyst, supporting materials, exchanger, molecular sieves etc.

6. PROPOSED WORK PLAN:

The present work aimed to synthesize silica based mesoporous materials from industrial and agricultural wastes and their applications in various physical and chemical perspectives is broadly elaborated in three consecutive years is depicted as follows:

First Year:-

1. To collect the references in relevant field.
2. To attend the seminars/ conferences/ workshops on relevant topic.
3. To study the various papers and finalize one of the schemes of synthesis such as hydrothermal or in *situ* method.

Second Year:-

1. To study the existing and new techniques which will be applied in our research work.
2. Collection of industrial and agricultural wastes such as coal fly ash, rise husk ash or pea nut husk or cane sugar husk followed by their calcinations so as to remove the carbons and magnetic material by magnetic stirring.
3. Screening of this calcite and demagnetized raw material to obtain fine residue for our further investigation.
4. Some characterization such as XRD, FTIR, SEM and EDAX will be taken to find out the percentage of silica and other components.
5. Rich silica raw material will be choose for our further synthesis of mesoporous siliceous material.
6. Silica will be leached by the leaching process from sodium hydroxide which will be a supernatant for the synthesis purpose.

7. Keeping the aim of synthesizing SBA-36, 48 from among the various mesoporous materials.
8. Synthesis process will be optimized by studying the effect of synthesis temperature, calcinations temperature, pH, time, effect of NaOH, reusability, etc.
9. To optimize the superior industrially optimum material, it is to be characterized by series of large characterization such as XRD, FTIR, EDAX, SEM, TEM, TGA and DTA, UV, etc.
10. Once the material is optimized, to enhance its physical and chemical activity post modification with some metal ion will be followed to prepare M-MCM 41, 48.
11. Post modified synthesized material will be used in some physical and chemical applications.

Third Year:-

1. To publish the work in reputed peer reviewed international journals.
2. To compile the data for thesis writing and eventually the thesis will be submitted to the concerned authority.

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